भारतीय मानक
Indian Standard

IS 2600 (Part 7) : 2022

डाई कॉस्टिग के लिए जिंक एवं जिंक आधारित मिश्रणों के रासायनिक विश्लेषण की विधियाँ

भाग 7 टाईट्रिमेट्रिक विधि द्वारा एल्यूमिनियम ज्ञात करना

(पहला पुनरीक्षण)

Methods of Chemical Analysis of Zinc and Zinc Base Alloys for Die Castings

Part 7 Determination of Aluminium by Titrimetric Method

(First Revision)

ICS 77.120.60

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 7) (First Revision) was adopted by Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Metals Sectional Committee had been approved by Metallurgical Engineering Division Council.

This standard was first published in 1964 and has been revised (in parts) on the basis of implementation of the standard by the manufacturers and the testing.

This standard (Part 7) cover the methods for the determination of aluminium by titrimetric method in zinc and zinc based alloys for die castings. The other parts in the series are:

Part 1	Determination of copper, iron, nickel, tin and thallium by spectrophotometric,
Part 2	Determination of copper, iron, lead and cadmium by atomic absorption spectrometric method,
Part 3	Determination of indium by atomic absorption spectrometric method,
Part 4	Determination of chromium by spectrometric method,
Part 5	Analysis by inductively coupled plasma optical emission spectrometry, and
Part 6	Determination of magnesium by atomic absorption spectrometric method.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 7 DETERMINATION OF ALUMINIUM BY TITRIMETRIC METHOD

(First Revision)

1 SCOPE

This standard (Part 7) covers the method for determination of aluminum by titrimetric method for the analysis zinc and zinc alloys in range as specified in IS 209, IS 713 and IS 742.

2 REFERENCE

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subjected to revisions, and parties to agreement based on these standards are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

IS No.	*Title*
209 : 1992	Zinc ingot — Specification (*fourth revision*)
265 : 2021	Hydrochloric acid (*fifth revision*)
713 : 1981	Zinc based alloy ingot for die casting (*second revision*)
742 : 1981	Zinc based alloy die casting (*second revision*)
1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis

3 SAMPLING

The samples shall be drawn and prepared in accordance with the procedure laid down in IS 1817.

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified, analytical grade reagents and distilled or demineralized water shall be employed in the test.

4.2 Reagents

4.2.1 *Hydrochloric Acid*, rd = 1.16 (*see* IS 265).

4.2.2 *Nitric Acid*, rd = 1.4.

4.2.3 *Hydrochloric Acid* (1 + 1) — Mix one volume of hydrochloric acid (*see* **4.2.1**) with one volume of nitric acid (*see* **4.2.2**).

4.2.4 *Hydrogen Peroxide*, 30 Percent Solution — Mass fraction.

4.2.5 *Hydroxylammonium Chloride* — Dissolve 200 g of hydroxylammonium chloride in one liter water.

4.2.6 *Ethylenediamine Tetra — Acetic Acid Disodium Salt* (EDTA) — Dissolve 65 g of ethylenediamine tetra-acetic acid disodium salt in one liter water.

4.2.7 *Ammonia solution*, rd = 0.91 g/ml.

4.2.8 *Sodium Fluoride, Saturated Solution* — Dissolve 60 g of sodium fluoride in 1 litre of boiling water. Cool to room temperature and filter.

4.2.9 *Sodium Acetate, Buffer Solution* — Dissolve 135 g of sodium acetate ($CH_3COONa.3H_2O$) in approximately 300 ml of water. Add 13 ml of glacial acetic acid (*see* **4.2.15**). Check that the *p*H is within the range 5 to 5.5. Make up the volume to 500 ml with water.

4.2.10 *Standard Zinc Solution*, 0.05 M — Dissolve 3.269 g of high purity zinc in 40 ml of hydrochloric acid (1 + 1) (*see* **4.2.3**) in a 250 ml beaker fitted with a watch-glass. Dilute with 100 ml of water. Add 2 drops of methyl red solution (*see* **4.2.11**). Neutralize with ammonia solution (*see* **4.2.7**). Add hydrochloric acid (1 + 1) (*see* **4.2.3**) drop by drop until the color changes to red. Transfer quantitatively to a 1 litre volumetric flask. Add 5 ml of hydrochloric acid (1 + 1) (*see* **4.2.3**), dilute to the mark with water and mix.

4.2.11 *Methyl red - Ethanolic Solution* — Dissolve 0.02 g of methyl red in 100 ml of ethanol.

4.2.12 *Xylenol Orange 10 g/l Solution*

4.2.13 *Aqua Regia* — Mix 3 volumes of hydrochloric acid (*see* **4.2.1**) with 1 volume of nitric acid (*see* **3.2.2**).

4.2.14 *Xylene Cyanol — 1 g/l solution.*

NOTE — The use of this reagent is optional.

4.2.15 *Glacial Acetic Acid*, rd = 1.05 g/ml.

5 PROCEDURE

5.1 Preparation of Sample Solution

Weigh 5 g of the sample to the nearest 0.001 g. Transfer the sample into a 500 ml beaker and cover with a watch glass, and dissolve by carefully adding 50 ml of the hydrochloric acid (1 + 1). Oxidize and complete the dissolution by adding a few drops of hydrogen peroxide (*see* **3.2.4**).

5.2 Add 5 ml of hydroxylammonium chloride solution (*see* **4.2.5**) to decompose the excess of hydrogen peroxide. Heat the solution carefully.

Dilute to 100 ml with water and cool to room temperature. Transfer quantitatively.

a) for Al contents (mass fractions) ≤ 15 percent : to a 250 ml volumetric flask; and
b) for Al contents (mass fractions) > 15 percent : to a 1000 ml volumetric flask.

Dilute to the mark with water and mix.

5.3 Transfer a 25 ml aliquot to a 500 ml conical flask.

5.4 Add successively:

a) approximately 100 ml of water.
b) EDTA solution:
    1) For Al contents (mass fractions) ≤ 15 percent: 55 ml of EDTA solution (*see* **4.2.6**); and
    2) For Al contents (mass fractions) > 15 percent: 25 ml of EDTA solution (*see* **4.2.6**).
c) 5 drops of methyl red solution (*see* **4.2.11**).

NOTE — Bromothymol blue may be used as the indicator for neutralization instead of methyl red.

5.5 Neutralize exactly with ammonia solution (*see* **4.2.7**) until the colour changes to yellow. Add 25 ml of buffer solution (*see* **4.2.9**).

5.6 Boil for 2 min to 3 min. Cool to room temperature.

5.7 Add 2 to 3 drops of xylenol orange solution (*see* **4.2.12**).

NOTE — For some operators, the end-point can be made even more readily detectable by the addition of not more than 1 ml of xylene cyanol solution (*see* **4.2.14**).

5.8 Titrate the excess of EDTA with standard zinc solution (*see* **4.2.10**), until the colour changes to purple.

5.9 Add 25 ml of sodium fluoride solution (*see* **4.2.8**). Boil for 2 min to 3 min. Cool to room temperature.

5.10 Titrate the liberated EDTA with the standard zinc solution (*see* **4.2.10**), until the colour changes to purple.

5.11 Calculation

The aluminium content shall be given, as a percentage by mass:

a) For Al contents (mass fractions) ≤ 15 percent: by the formula $V \times 0.269\,8$; and
b) For Al contents (mass fractions) > 15 percent: by the formula $V \times 1{,}079\,2$; where V is the volume, in millilitre, of standard zinc solution used to titrate the liberated EDTA.

If a number of determinations are carried out, then the mean of all the results shall be calculated.

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Amendments Issued Since Publication

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